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13. ABSTRACT (Maximum 200 Words) Pairs of molecules that cocrystallize to form predictable noncentrosymmetric packing patterns have been identified. These systems, termed quasiracemates, consist of isosteric molecular components that are chemically unique, but differ in chirality. The direction of these studies centered on establishing factors that influence quasiracemate formation by inspection of crystallographic data of full sets of related crystals. Design strategies for these systems included molecular components with functional groups that varied in electronic and steric properties. Various molecular pairs were synthesized and comparative studies of crystallographic data allowed assignment of the role of molecular environment on quasiracemate formation. Unlike the enantiomeric structures, the "true" racemates mimic the quasiracemate structures. The alignment of quasiracemate building blocks in predictable approximately centrosymmetric assemblages stems from the propensity for organic compounds, in general, to form centrosymmetric arrangements in crystals. This topological effect was demonstrated and exploited as a tool to fabricate a wide variety of molecular systems with rigorously noncentrosymmetric crystal motifs.					
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**FINAL REPORT FOR: APPROXIMATE SYMMETRY IN THE DESIGN
OF NON-LINEAR OPTICAL MATERIALS**

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2. OBJECTIVES

The major research objectives of this program are:

- to investigate the role of approximate symmetry as a reliable tool towards crystal engineering;
- to develop new nonlinear optical (NLO) materials based on symmetry-directed aggregation; and
- to develop a formal collaborative agreement between a historically black (HBCU) and a research-intensive doctoral degree-granting institution

3. STATUS OF EFFORT

Pairs of molecules that cocrystallize to form predictable noncentrosymmetric packing patterns have been identified. These systems, termed quasiracemates, consist of isosteric molecular components that are chemically unique, but differ in chirality. The direction of these studies centered on establishing factors that influence quasiracemate formation by inspection of crystallographic data of full sets of related crystals. Design strategies for these systems included molecular components with functional groups that varied in electronic and steric properties. Various molecular pairs were synthesized and comparative studies of crystallographic data allowed assignment of the role of molecular environment on quasiracemate formation. Unlike the enantiomeric structures, the "true" racemates mimic the quasiracemate structures. The alignment of quasiracemate building blocks in predictable approximately centrosymmetric assemblages stems from the propensity for organic compounds, in general, to form centrosymmetric arrangements in crystals. This topological effect was demonstrated and exploited as a tool to fabricate a wide variety of molecular systems with rigorously noncentrosymmetric crystal motifs.

4. ACCOMPLISHMENTS/NEW FINDINGS

The goal of this section is not to provide an exhaustive compilation of data that is sufficiently published or submitted, but rather to show how the series of studies has provided a strategy

towards the fabrication of materials with rigorously noncentrosymmetric packing motifs necessary for materials applications with optical nonlinearities. Many of these goals were met, and the paragraphs below will show that methods are now available to form predictable pseudocentrosymmetric molecular assemblages by use of molecular shape mimicry. The studies performed to date provide a knowledge base that allows for the engineering of interactions in molecular crystals towards the optimization bulk property materials.

4.1 Quasiracemate Design Strategies: Propionic Acid Templates

Because the properties of molecular solid-state materials relate intimately to their molecular arrangements, an understanding of the factors that influence weak intermolecular interactions is an important priority in the chemical sciences today. Due to the detail of information obtained from single-crystal X-ray structures, one of the potentially most fertile grounds in which to study the nature and effects of such interactions is in molecular crystals. A major challenge in the understanding of molecular arrangements in crystals is that these structures are determined by the combination of many weak and ill-defined intermolecular forces. While many of the principles responsible for packing motifs in crystals have been elucidated, a unified set of criteria that describes the intra- and intermolecular interactions remains undiscovered. This lack of predictive power has been countered somewhat by designing molecules with functional groups that provide directionality. In the last few decades, strategies toward the design of materials with predetermined molecular arrangements have successfully exploited the existing database of known structural biases (*e.g.*, hydrogen bonds, halogen-halogen interactions and inclusion phenomena) to produce predictable discrete patterns and secondary structural motifs such as 1D tapes and columns. Although recent advances show great promise for the field of materials science, the "complete" prediction and control of molecular patterns by the assembly of secondary structures into tertiary patterns based solely on molecular structure still remains elusive.

Steps toward the optimization of bulk properties require tailoring of intermolecular orientations as with organic materials with second-order nonlinear optical (NLO) properties. NLO devices have various applications in the development of frequency doubling and optical switching materials. A basic tenet of studies that fabricate NLO molecular devices center on

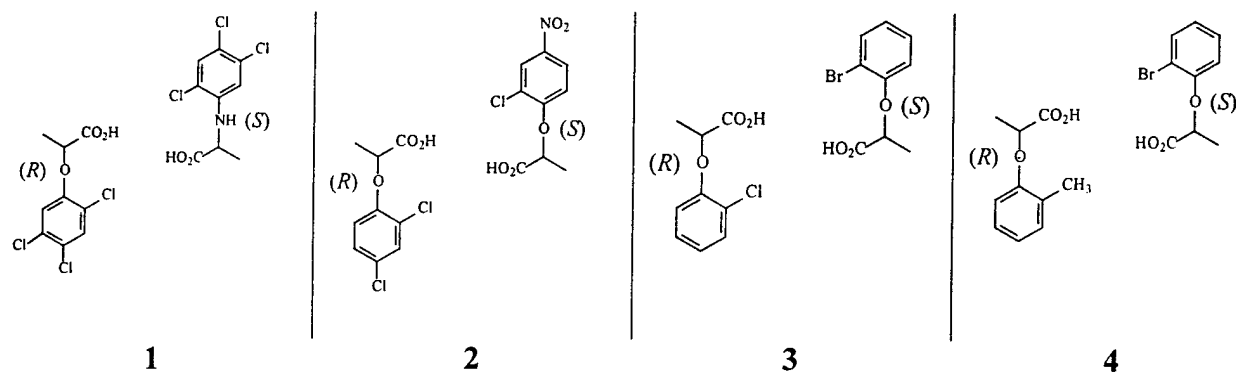
systems that exhibit fast response times to external field stimuli (electronic susceptibility, $\chi^{(2)}$) and large hyperpolarizabilities (β and γ). Such prerequisites translate to organic materials with highly polarizable molecules and noncentrosymmetric molecular arrangements. Even though the correlation between chemical and crystal structure to NLO behavior is somewhat definable, the propensity of organic materials to arrange in centrosymmetric patterns precludes the majority of organic compounds as candidates for NLO studies.

The importance of understanding the factors that control molecular assemblages that lead to a particular motif in crystals is undisputed; even so, attention to structural features that mitigate against the formation of desired molecular architectures can provide equally important information. For example, numerous studies employ carboxylic acid groups to create predictable intermolecular contacts. The universal acceptance of the $-\text{CO}_2\text{H}$ moiety as a viable structural feature for constructing molecular scaffolds stems from the substituent's ability to form strong and directional connections with a wide variety of functional groups. Molecular patterns of simple organics generated by synplanar $-\text{CO}_2\text{H}$ geometries frequently lead to homodimers linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds of graph set notation $R_2^2(8)$. Unlike the pattern and abundance of crystal structures that possess the synplanar $-\text{CO}_2\text{H}$ conformer, the antiplanar geometry of carboxylic acids, which allows $C(4)$ hydrogen-bonded chains, occurs to a much lesser extent. In the past, the formation of these catemers appeared unexpectedly, but more recent accounts of the motif describe several structural features that influence the pattern and rarity of its occurrence. Even with this additional information, the catemer motif lacks a high degree of predictability, and we are not aware of any studies that strategically employ the antiplanar conformation of carboxylic acids as a structural tool for materials design. With time, and with the elucidation of additional relevant crystal structures, the factors that influence this structural pattern will most likely be elucidated and possibly turned to advantage, much like the previously ill-defined $\text{C}-\text{H}\cdots\text{O}$ interactions.

Molecular shape is well recognized in general terms as a significant contributor to crystal packing, but the detailed degree to which these topological factors influence molecular arrangements remains relatively vague. The propensity of organic materials to form centrosymmetric arrangements has recently been described, and is most evident by the >50% occurrence of organic structures in the space groups $P2_1/c$ and $P\bar{1}$. Our investigations of the

importance of centrosymmetry utilize materials that contain two components, (*R*)-X and (*S*)-X'; where X and X' represent sterically similar molecules. This similarity can be achieved by substitution of groups that are sterically similar, though they may be electronically quite different. Such materials are termed quasiracemates. Unlike the majority of crystal engineering studies that use hydrogen bonds to produce desirable packing motifs, these cocrystalline systems do not depend primarily on the control of complementary electrostatic interactions or lock-and-key relationships of conventional host-guest systems. The direction of these studies centers on establishing factors that influence quasiracemate formation by inspection of crystallographic data of full sets of related crystals. These studies provide not just the structure of the quasiracemate, but also structures of the individual enantiomers and of the "true" racemates (Figure 1). The structural patterns represented for an entire family of five structures provide a systematic method for establishing structural relationships.

The self-assembly of approximate centrosymmetrically related materials in the crystalline state was investigated by a comparative study of a homologous series of phenoxypropionic acid derivatives, 1 – 4. The selection of sterically similar but chemically unique functional groups for this study (i.e., O/NH, Cl/NO₂, Br/Cl and CH₃/Br) provides evidence for the formation of quasiracemates and reveals the breadth of utility of this design strategy.



The crystal structures of the quasiracemate components of 1 are shown in Figure 1. Inspection of the crystal structures of this structural family reveals distinct patterns organized by hydrogen bonds. Inspection of the crystal structures of the racemates (Figure 1, bottom l and r) form the expected carboxylic acid head-to-head dimer linked by O–H···O contacts. Inspection of the structures shows no evidence for significant Cl···Cl contacts or face-to-face π -stacking and thus hydrogen bonding represents the principal noncovalent controlling feature of these

$R_2^2(8)$ centrosymmetric assemblies. Surprisingly, the packing motif of (\pm)-anilino derivative (Figure 1, bottom l) reveals N \cdots O contacts > 3.3 Å suggesting the absence of N–H \cdots O stabilization.

A comparison of the packing motifs of the racemic and enantiomeric components of **1** reveals significant differences in hydrogen bond topologies. In contrast to the centrosymmetric patterns observed for the racemic structures, molecules of (*R*)-phenoxy are linked by O–H \cdots O interactions to form extended $C_1^1(4)$ hydrogen bonded chains along the *a*-axis (Figure 1, top r). Unlike the (\pm)-phenoxy, the structure of (*S*)-anilino is further stabilized by weak N–H \cdots O hydrogen bonds (Figure 1, top r). Similar to the racemates of **1**, the crystal structure of (*R*)-phenoxy reveals pairs of molecules associated by O–H \cdots O contacts to form $R_2^2(8)$ patterns. Since both molecules that comprise the pattern possess identical stereoconfigurations no centrosymmetric relationship exists between the two molecules. In fact, the pattern is generated by two symmetry independent (*R*)-phenoxy molecules.

The formation of quasiracemate **1** by cocrystallization of anilino (*S*)-anilino and (*R*)-phenoxy is evident by inspection of the Figure 1 (bottom ctr). Association of the two components in the crystal results in an approximate centrosymmetric pattern that closely mimics the previously described patterns for racemates. The relationship of these structures may be described as isostructural due to the similarity in hydrogen bond and packing patterns as well as unit cell constants.

In addition to the systematic study of the crystal structure patterns and properties, the calculation of packing energies for the racemates and quasiracemate of **1** provides further evidence of the factors that control the cocrystallization of the quasiracemate. A comparison of the structural parameters in Table 1 attests to the isostructural relationship of the molecular components. Even so, the similarity of packing energies provides additional evidence and the ability of parameterize the isostructural relationships of the related compounds. Calculation of the packing energies of the phenoxy and anilino racemates reveals energies of -250 and -272 kJ/mol for pair of molecules. Such energies (E_{pack}) compare closely to that of the quasiracemate (-256 kJ/mol) and indicate an energetically similar quasiracemate. By substituting one of the components of the quasiracemate with the other component in the quasiracemate structure, one

can simulate the structure of the "true" racemate. The energies (E_{sim}) based on these calculations support quasiracemate formation as controlled by energetically favorable molecular arrangements. In this system, the quasiracemate structures mimic the racemate structures as a result of the isosteric nature of the chemically different groups.

In a similar manner the crystal structures of the enantiomers, racemates, and quasiracemates for systems 2 – 4 were determined. A comparative study of each system provided evidence for the identification of structure-property relationships. In all of the cases, a strong structural correlation exists between the racemates and quasiracemate - presumably directed by the effects of molecular shape mimicry.

As shown in Figure 2, the crystal structures of the enantiomeric and racemic propionates of 2 reveal structural patterns analogous to the collection of structures for 1. The structures of both (*R*)-dichloro and (*S*)-chloronitro derivatives show molecules linked by O–H···O interactions to form extended $C_1^1(4)$ hydrogen bonded chains (Figure 2, top). Although the difference in unit cell parameters for these structures precludes an isostructural assignment, the topological patterns of the overall structures are strikingly similar. Unlike the enantiomers, molecules in the racemate structures form predictable centrosymmetric dimers associated by O–H···O contacts to form $R_2^2(8)$ patterns. Crystallization of a 1:1 CH_2Cl_2 solution of (*R*)-dichloro and (*S*)-chloronitro moieties by slow evaporation yields crystals of quasiracemate 2. A comparative study of the crystallographic data sets and unit cell constants of the racemic and quasiracemic materials reveals an isostructural relationship.

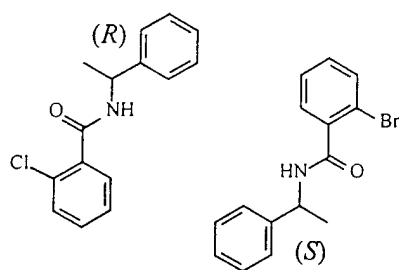
Quasiracemate systems 3 – 4 differ only in the Cl, Br, and CH_3 functionalities. These groups are chemically unique but possess similar size and shape characteristics (group volumes: Cl, 19.6 \AA^3 ; Br, 27.6 \AA^3 ; CH_3 , 20.7 \AA^3) and thus represent a viable alternative to the previously studied quasiracemate systems 1 – 2. For both systems the enantiomeric components needed to carry out quasiracemate formation were synthesized, but suitable crystals for X-ray diffraction studies could not be obtained from standard crystal growth techniques. Once again, from inspection of the racemic structures (Figure 4, bottom l and r), the similarity of molecular arrangements and unit cell parameters reveals a isostructural relationship. This underlying theme of crystal structure similitude (approximately centrosymmetric) of the racemic and quasiracemic materials is certainly evident as revealed from these systematic examples; however, as shown with system

4 the quasiracemate structure may not be isostructural to the racemates. Figure 4 shows the crystal structures of the CH_3 and Br derivatives, with molecules linked to form predictable centrosymmetric dimers associated by $\text{O}-\text{H}\cdots\text{O}$ contacts to form $R_2^2(8)$ patterns. The similarity of these carboxylic acid dimer units does not propagate to the gross packing motif of these systems. Inspection of the (\pm)-methyl crystal structure reveals four symmetry independent molecules that crystallize in space group $P\bar{1}$ and show little semblance to the analogous quasiracemate or bromo derivative of system 4. The consequence of this observation is of little value since the existence of polymorphs can not be ruled out.

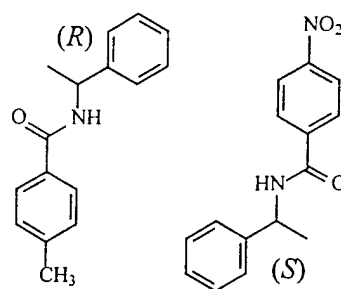
In each case described, the relationship between the two chemically different molecules of the quasiracemate approximates the centrosymmetric symmetry operator. The two chemically unique molecules that constitute the quasiracemate mimic the discrete patterns found in the "true" racemate structures and are influenced by the dominant packing motif described by the inversion symmetry operator.

4.2 Extending Design Strategies: Amide templates

The design of the systems studies thus far centers on a homologous series of propionic acid derivatives. In order to test the utility of quasiracemate formation the program investigated a series of unrelated amide quasiracemic systems. Unlike the carboxylic acid quasiracemic systems 1 - 4 described previously, systems 5 - 6 possess a significantly different molecular framework by the addition of the secondary amide functionality. Although prior considerations to this study acknowledged the differing influence of the carboxylic acid (dimers) and secondary amide (infinite chains) groups on molecular arrangements, the effect of designing molecules with amide frameworks for the formation of quasiracemic materials had not previously been studied. Additionally, quasiracemate 5 builds upon the results of the programs previous studies by use of Cl/Br functionalities, but deviates considerably in 6 by use of CH_3/NO_2 groups that lack isosteric and isoelectronic relationships.



5



6

Inspection of Figures 5 and 6 shows the crystal structures of the enantiomeric, racemic, and quasiracemic compounds **5** and **6** with hydrogen bond patterns characteristic of molecules with secondary amide functionalities. The structures of the enantiomeric and racemic components of both systems show molecules linked by N-H \cdots O interactions to form extended $C_1^1(4)$ hydrogen bonded chains. Unlike systems **1** – **4**, the molecular patterns represented in the structures lack isostructurality and may be most evident by inspection of unit cell dimensions and structural architectures in Figures 5 and 6. Although the difference in unit cell parameters for these structures precludes any strict isostructural assignment, the topological patterns of the overall structures are strikingly similar. A 1:1 cocrystallization of the appropriate enantiomers produced crystals consisting of equal portions amide building-blocks. Analysis of these quasiracemic materials (**5** and **6**) by crystallographic methods yielded the structures shown in Figures 5 and 6. Although these structures do not mimic those of the corresponding racemates the quasiracemates form pseudocentrosymmetric patterns analogous to the racemate structures.

In conclusion, this work has led to a series of highly predictable noncentrosymmetric materials by designing compounds to be isosteric and to favor cocrystallization in approximately centrosymmetric arrangements as R -X and S -X' quasiracemic crystals. The design of crystals that form quasicentrosymmetric arrangements hinges on the propensity of organic materials to crystallize in centrosymmetric space groups. The program efforts in this area have been successful at implementing this structural bias by fabricating and determining the structures of several quasiracemic systems. The success of these studies in forming quasiracemic materials follows a rational strategy: the two components that comprise the quasiracemate system are designed to (i) be isosteric, and (ii) possess hydrogen-bond donor and acceptor groups to aid in

crystal growth, but (iii) differ in chemical composition, and (iv) differ in chirality. Factors that govern the formation of quasiracemic assemblages stems from the propensity of organic molecules to crystallize in (at least approximately) favorable centrosymmetric arrangements.

Figure 1. Graphical representation of crystallographic strategies for quasiracemate studies.

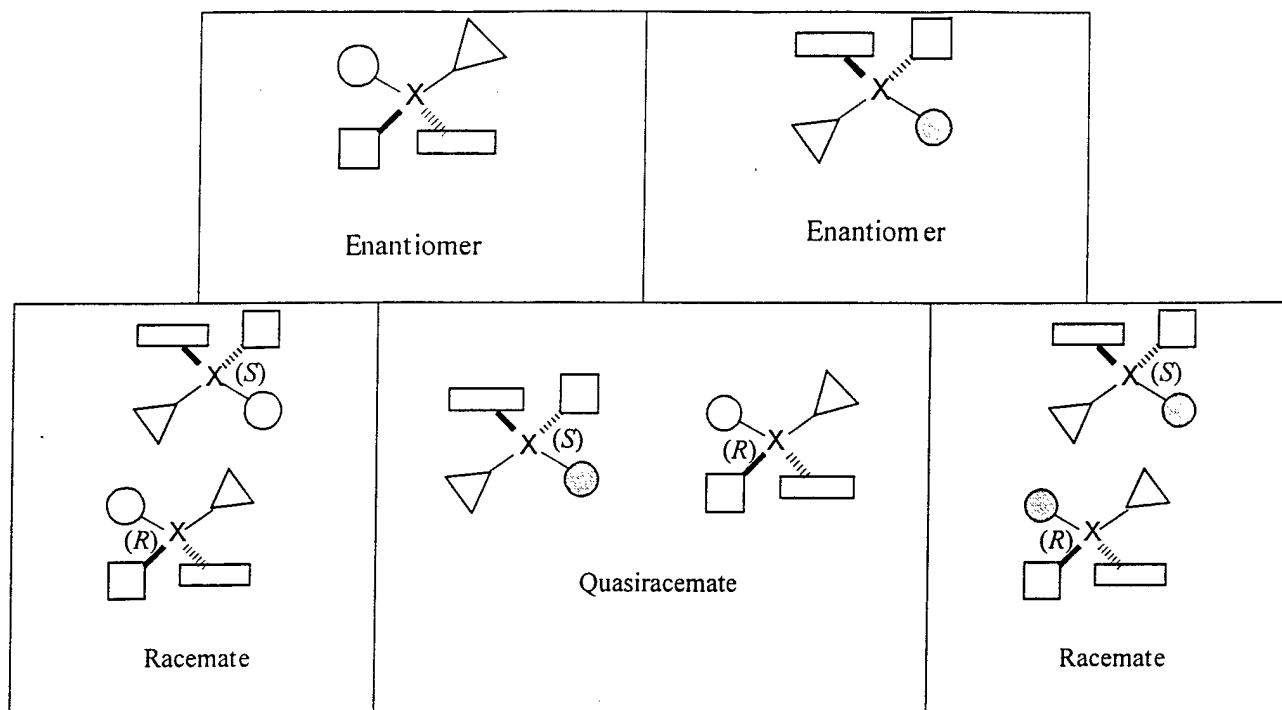


Figure 2. Crystal structures of the enantiomers, racemates, and quasiracemates of 1.

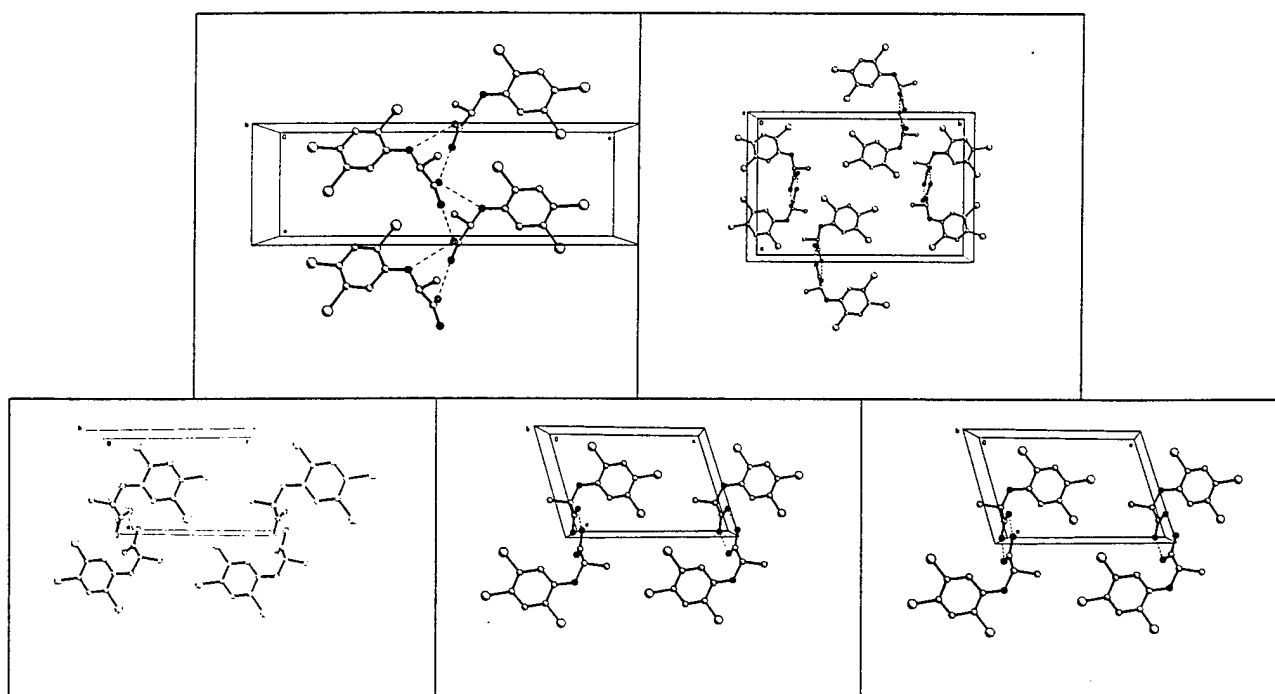


Figure 2. Crystal structures of the enantiomers, racemates, and quasiracemates of **2**.

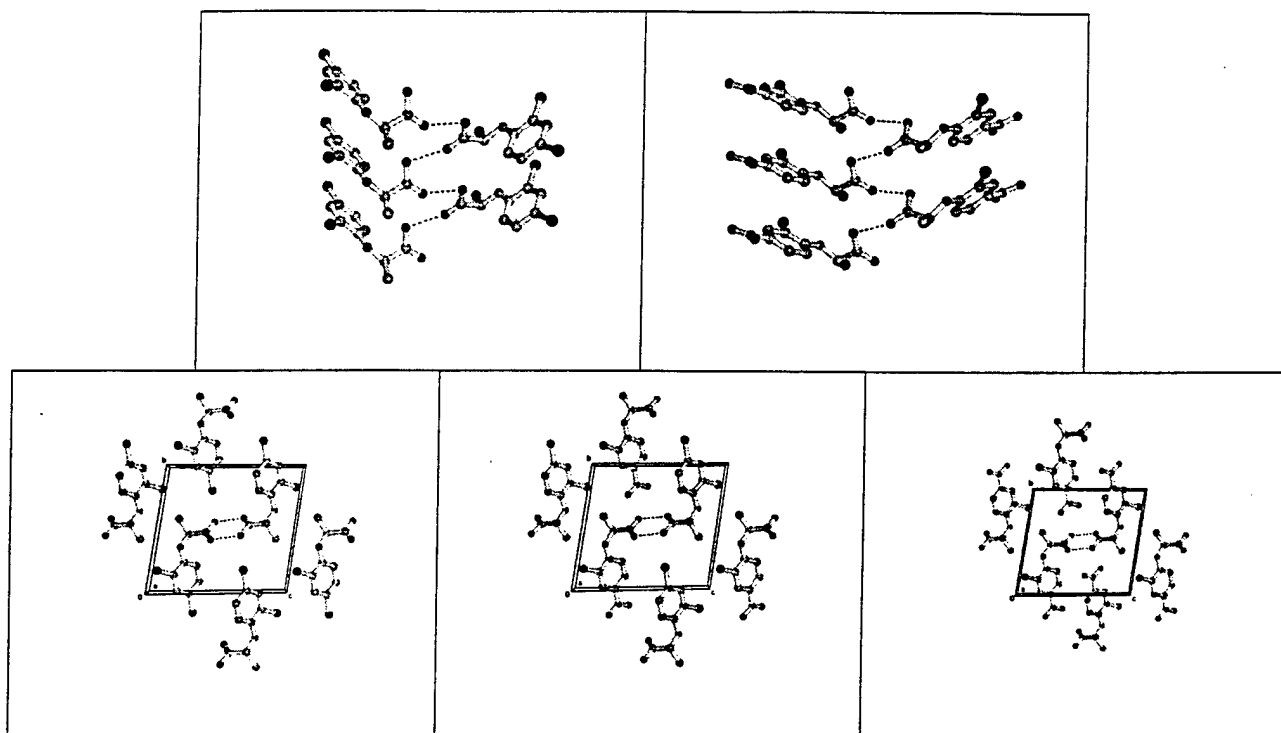


Figure 3. Crystal structures of the enantiomers, racemates, and quasiracemates of **3**.

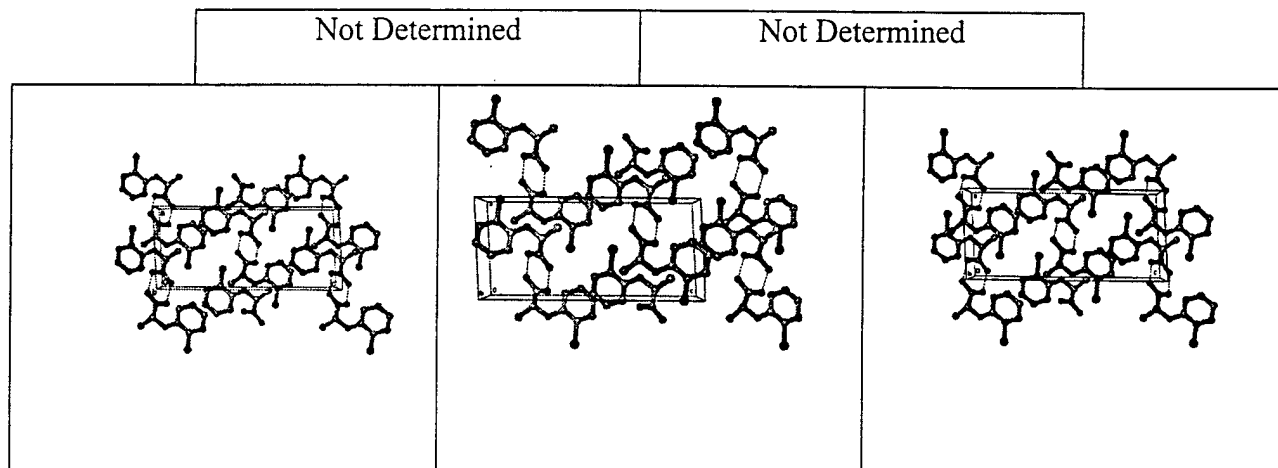


Figure 4. Crystal structures of the enantiomers, racemates, and quasiracemates of 4.

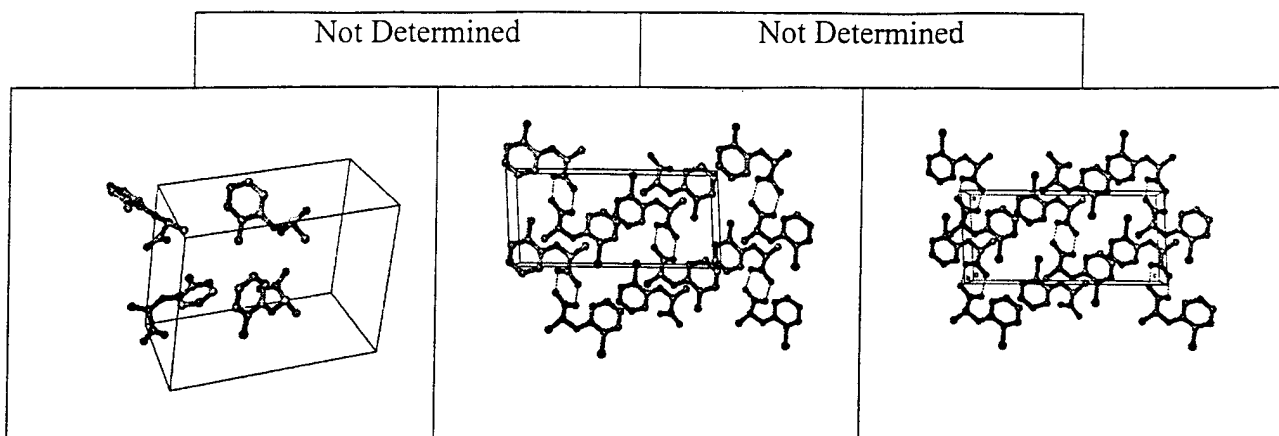


Figure 5. Crystal structures of the enantiomers, racemates, and quasiracemates of 5.

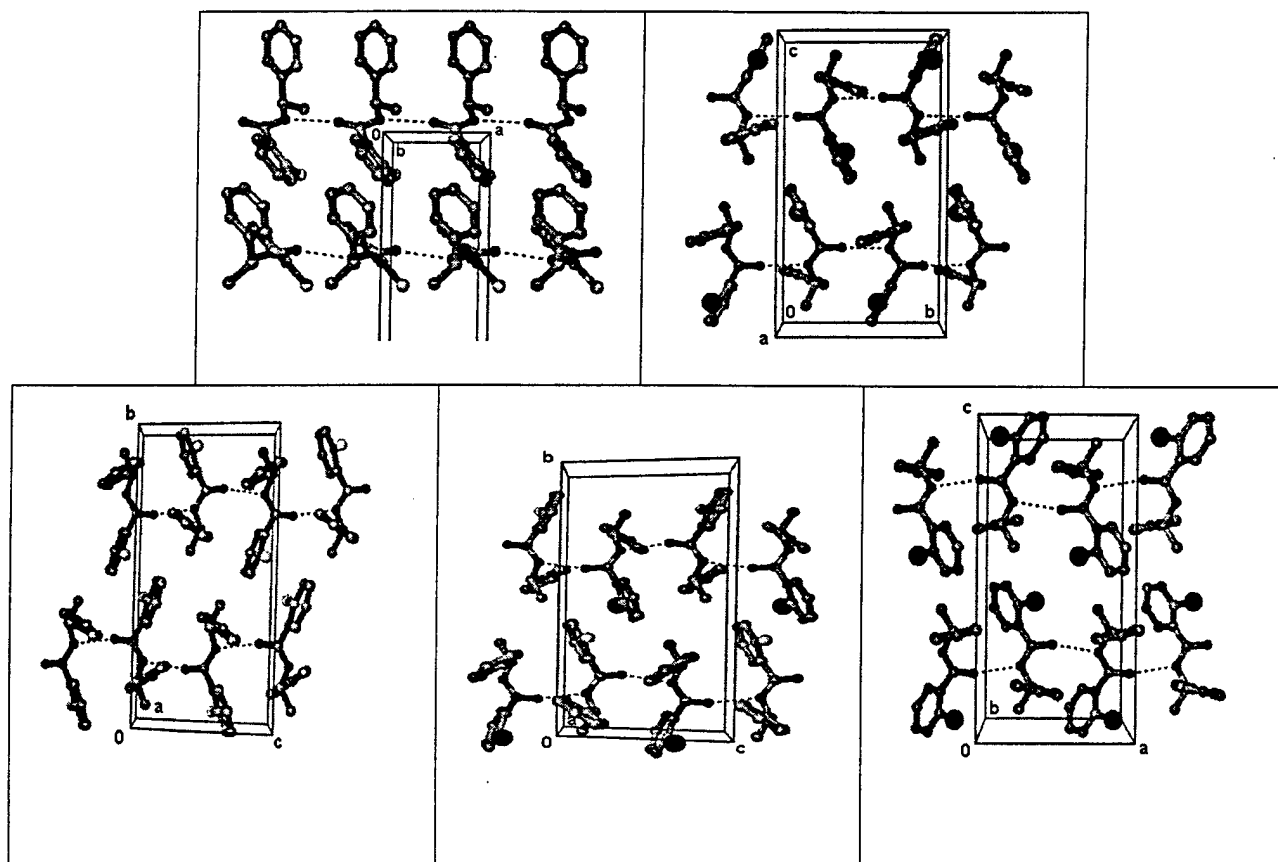


Figure 6. Crystal structures of the enantiomers, racemates, and quasiracemates of 6.

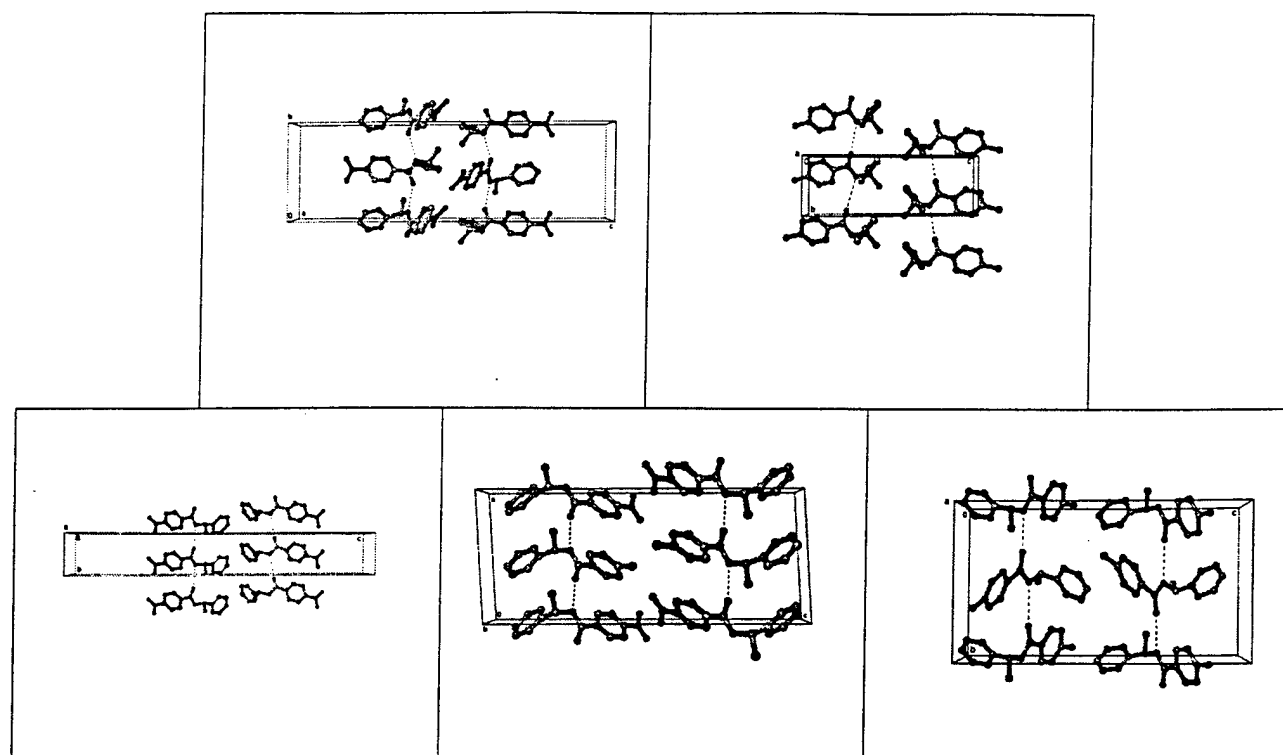


Table 1. Unit cell constants and molecular modeling packing energies for quasiracemate 1.

Parameter	(±)-Phenoxy	Quasiracemate	(±)-Anilino
<i>a</i>	4.472	4.562	4.559
<i>b</i>	10.536	10.468	10.556
<i>c</i>	11.426	11.366	11.411
α	80.61	80.31	80.74
β	82.07	82.19	82.57
γ	82.45	82.38	82.35
<i>V</i>	522.8	526.6	533.9
Space Group	$P\bar{1}$	$P1$	$P\bar{1}$
R-value	0.050	0.058	0.053
E_{pack}	-250 (kJ/mol)	-259	-272
E_{sim}		-259	-256

5. PERSONNEL SUPPORTED

Hernan Aldaz-Palacios

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Shella Fomula

Mukta S. Hendi

Paul Hooter

Barbara Ipaud

Ning Leh-Chang

Tricia McCampbell

Thomas Otto

Kraig A. Wheeler

6. PUBLICATIONS

1. "Pseudocentrosymmetrically Programmed Materials," *Crystal Engineering*, Submitted, K. A. Wheeler, M. Hendi, S. Fomulu, N.-L. Chang, and R. E. Davis.
2. "Spontaneously Resolved Chiral Arylsulfonamides," *Crystal Engineering*, Accepted, K. A. Wheeler, M. Hendi, and R. E. Davis.
3. Raymond E. Davis, James K. Whitesell and Kraig A. Wheeler; Molecular Shape as a Design Criterion in Crystal Engineering; invited chapter in "Implications of Molecular and Materials Structure for New Technologies," NATO ASI Series, Editors J. A. K. Howard and F. H. Allen; 1999, 263-274.

7. INTERACTIONS

7.1 Papers presented at Scientific Conferences

1. "Organised Molecular Assemblies By Shape Mimicry" American Crystallographic Association Meeting, St. Paul; **2000**, K.A. Wheeler, M. Hendi, S. Fomulu, and R.E. Davis.
2. "Some Limitations in Attempted Quasiracemate Formation" American Crystallographic Association Meeting, St. Paul; **2000**, R.E. Davis, K.A. Wheeler, M. Hendi, and S. Fomulu.
3. "Strategies and Limitations of Molecular Shape Directed Packing Motifs" Eleventh Annual Midwest Organic Solid State Chemistry Symposium, West Lafayette, IN, June **2000**. S. Fomulu, M. Hendi, R.E. Davis, and K.A. Wheeler
4. "Molecular Shape Directed Aggregation of Sulfamoylbenzamide Derivatives Towards Structure Prediction" 2nd Annual Undergraduate Research Symposium at University of Maryland Baltimore County, Baltimore, MD, **1999**, T. McCampbell, M. Hendi, and K.A. Wheeler.
5. "Approximate Symmetry As A Tool To Generate Molecular Motifs" P11.0D.010, International Union of Crystallography, Glasgow, UK, July **1999**. K. A. Wheeler, M. Hendi, S. Fomulu, and R.E. Davis.
6. "Approximate Symmetry in Crystal Engineering: Packing Energies of Racemates and Quasiracemates" P11.0D.009, International Union of Crystallography, Glasgow, UK, July **1999**. R.E. Davis, Ning-L. Chang and K.A. Wheeler.
7. "Crystal Engineering: An Approximate Centrosymmetric Approach" Tenth Annual Midwest Organic Solid State Chemistry Symposium, Indianapolis, IN, June **1999**. E. Benjamin, M. Hendi and K.A. Wheeler.
8. "Crystal Engineering: A Centrosymmetric Approach" Invited presentation at Brandeis University, Waltham, MA, May **1999**. K.A. Wheeler.
9. "Ligand Dependent Site specific Association Of Two-Coordinate Ag(I):Pyridine Complexes" Experimental Biology National Meeting, Washington, DC, April **1999**; Wheeler, K. A and Carrington, D.M.

10. "Symmetry Directed Crystal Engineering" Midwest Organic Solid State Chemistry Symposium, Kansas State University, Manhattan, KS, 1998; Wheeler, K.A.; Hendi, M. S.; Fomulu, S. and Benjamin, E.
11. "Further Studies of Fredga's Quasiracemates" 1st Annual Undergraduate Research Symposium at University of Maryland Baltimore County, Baltimore, MD, 1998, Fomulu, S., Hendi, M., and K.A. Wheeler.
12. "Quasiracemates: Symmetry and Crystals" 1st Annual Undergraduate Research Symposium at University of Maryland Baltimore County, Baltimore, MD, 1998, Hooter, P., Hendi, M., and K.A. Wheeler.
13. "Approximate Centrosymmetry as a Tool for Crystal Engineering" E0206, American Crystallographic Association Meeting, Washington, D.C.; 1998, M. Hendi, S. Fomulu, E. Benjamin, K.A. Wheeler, R.E. Davis.

7.2 Consultive and Advisory Functions to other Laboratories

1. Advisory role provided on an ad hoc basis to the Applied Optics Center for the state of Delaware and AFOSR.

7.3 Transitions

1. Kraig Wheeler/Delaware State University, b. Nonlinear optical response materials, c. Applied Optics Center, N. Melikechi, 302-857-6656, d. Second harmonic generation output materials

8. NEW DISCOVERIES INVENTIONS OR PATENT DISCLOSURES

1. NA

9. HONORS/AWARDS

1. Kraig Wheeler, Recipient of the 2000 Excellence In Research & Creative Activity award at Delaware State University.